

(19)



Euroches Patentamt
European Patent Office
Office européen des brevets



(11)

EP 0 935 020 A1

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:
11.08.1999 Bulletin 1999/32

(51) Int Cl.⁸: **D21H 11/18, D21D 1/20,**
D21H 17/67

(21) Application number: **99660003.7**

(22) Date of filing: **11.01.1999**

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE
Designated Extension States:
AL LT LV MK RO SI

(72) Inventor: **Silenius, Petri**
08800 Kirknemi (FI)

(74) Representative: **Papula, Antti et al**
Papula Rein Lahtela Oy,
P.O. Box 981
00101 Helsinki (FI)

(30) Priority: **09.02.1998 FI 980296**

(71) Applicant: **Metsä-Serla Corporation**
02100 Espoo (FI)

(54) **Noil consisting of fibrils for use in paper manufacture, method for its production, and paper pulp and paper containing such noil**

(57) The invention relates to a noil consisting of fibrils for use in paper manufacture, to be mixed in the paper pulp. The noil is produced from cellulose fibre by

refining and consists of noil fibrils that, in respect of size distribution, mainly correspond to wire screen fraction P50, i.e. 50 mesh, and the noil constitutes 0.1 -15w-% of the paper pulp after mixing.

EP 0 935 020 A1

Description

[0001] The present invention relates to noil for use in paper manufacture, as defined in the preamble of claim 1. Moreover, the invention relates to a method for producing such noil and to paper pulp and paper containing it.

[0002] Today, the trend of development of paper products is increasingly determined by customers and legislative measures. The buyers of information paper want to economise on postage and reduce the amount of waste produced. Further, waste processing charges depending on weight have been imposed on packaging materials. Generally, it seems that energy taxes and environmental protection taxes are being added as an extra imposition to the price of paper products. For these reasons, paper buyers want paper products of a lower grammage which still meet high quality requirements.

[0003] In this application, information paper refers to different kinds of printable paper and cardboard, coated or uncoated, manufactured on paper and cardboard machines, e.g. printing paper and graphic cardboard.

[0004] Because of the general trend of development described above, there is a need to produce high-quality information paper using a reduced amount of raw material. When the grammage of paper is reduced, its opacity becomes a critical property. Opacity can be increased by increasing the filler content of the paper, but this generally reduces its strength properties. Therefore, there is a need to alter the paper structure in a way that allows important product properties to be maintained at the same time. For paper-based communication to remain competitive with respect to electric communication, further improvements in the impression quality of paper products are required. - These general trends of development impose very high requirements on the raw materials of paper and on paper production processes. In order to meet the requirements, intensive efforts have been made in recent times to further develop paper raw materials and their production processes.

[0005] Specification FI 931584 presents a composite product for use as filler in paper and a method for its manufacture, based on precipitation of calcium carbonate on the surface of cellulose fibres. The fibres are mainly whole chemical pulp fibres, individual microfibrils are only present on fibre surfaces.

[0006] From specification FI 953238, a paper filler is known that consists of porous aggregates formed by calcium carbonate particles precipitated on noil fibrils produced by refining from cellulose fibre and/or mechanical pulp fibre; as to their size distribution, the noil fibrils correspond to wire screen fraction P100. The filler gives the paper better optical and strength properties and a lower grammage than earlier calcium carbonate based fillers. For the manufacture of this product, calcium carbonate must be precipitated on the surface of noil fibrils.

[0007] The object of the present invention is to dis-

close a new type of noil for use in paper manufacture which is mixed with paper pulp and which meets the requirements specified above and is easier to manufacture than corresponding kinds of noil and/or fillers known before.

[0008] A specific object of the invention is to disclose a new type of noil that gives the paper better strength properties, especially a better tensile strength and a better interlaminar strength as well as a lower grammage than commonly known prior-art kinds of noil and/or filler and that is still easier and cheaper to manufacture than corresponding types of noil and/or filler known in prior art. It is therefore an object of the invention to disclose a new type of noil which, when used in paper manufacture, makes it possible to add to the paper an increased amount of pigments to improve the optic properties of the paper being produced, and which is easier to manufacture than prior-art types of noil and/or filler that meet corresponding criteria. A further object of the invention is to disclose a type of noil whose production does not require any large additional investments and which can be produced using equipment generally already existing in a paper factory.

[0009] Another object of the invention is to disclose a method for producing said noil.

[0010] A further object of the invention is to disclose a paper pulp containing said noil, to be used in the manufacture of information paper.

[0011] Yet another object of the invention is to disclose a paper type manufactured using said noil.

[0012] The invention is based on the unexpected observation made during investigations that paper having the desired optical, strength and grammage properties can be produced by mixing the paper pulp with noil produced by refining from pulp fibre, preferably chemical pulp fibre, consisting of noil fibrils that, in respect of size distribution, mainly correspond to wire screen fraction P50, the amount of noil forming 0.1 - 15 w-%, more preferably 0.5 - 10 w-%, most preferably 2 - 4.5 w-% of the paper pulp. A new feature in the invention is particularly the fact that noil, added to paper pulp, gives the paper the desired, improved strength and/or grammage properties. The desired optic properties can be achieved by adding required pigments into the paper pulp without impairing the strength properties of the paper.

[0013] Before, a corresponding type of noil has been used in the manufacture of transparent paper sorts, such as butter paper and baking paper. However, the amounts of noil used in these cases have been substantially larger than in the present application. Moreover, in the present application, the noil makes it possible to achieve expressly improved optic properties because an improved paper strength allows the use of an increased filler content; in transparent paper sorts, such optic properties have not been aimed at.

[0014] The noil of the present invention differs from the filler presented in specification FI 953238 in that, according to the present application, the desired strength

and grammage properties are achieved by using noil without precipitation of pigment on noil, in other words, the invention makes it possible to add the pigment into the pulp in the conventional manner, whereas in specification FI 953238 the pigment, i.e. calcium carbonate, is precipitated onto noil fibrils.

[0015] According to the present application, the noil can be mixed as such in the paper pulp; the pigment can be added into the paper pulp before the noil is mixed or after it has been mixed, or it may be added in conjunction with the mixing of the noil. The mixing can be performed e.g. in a machine tank or in some other suitable, e.g. separate mixer or container.

[0016] According to a preferred embodiment of the invention, the noil consists of noil fibrils mainly corresponding to wire screen fraction P100.

[0017] The amount of noil in the paper pulp is 0.1 - 15 w-%, preferably 0.5 - 10 w-%, most preferably 2 - 4.5 w-% of the pulp, calculated in terms of dry matter.

[0018] The mass ratio of pigment to noil is e.g. 0.1 - 20, preferably 0.2 - 10, most preferably 2 - 6. In this description, all mass ratio values have been calculated in terms of dry matter.

[0019] The noil is preferably produced by refining cellulose fibre to a Schopper number > 80, preferably to a Schopper number between 80 - 90, or even to a higher Schopper number value.

[0020] In addition to the noil of the invention, conventional pigments, e.g. kaolin, talcum, titan dioxide, ground calcium carbonate, precipitated calcium carbonate (PCC), chalk, synthetic silicate (such as aluminium silicate, aluminium magnesium silicate), barium sulphate, aluminium hydroxide or in general any pigment, can be used in the paper pulp. Mixed and/or composite pigments can also be used. Different calcium carbonates and kaolin are especially well suited for this purpose. Particularly good results have been achieved using precipitated and ground calcium carbonates, e.g. calcium carbonates having a scalenohedral crystal structure.

[0021] The consistency of the noil is preferably of the order of 0.4 - 10 w-%.

[0022] The noil of the invention can be advantageously produced from cellulose fibres by refining so that the edge load is of the order of 0.1 - 8 Ws/m. The refining can be effected using any known type of refiner, e.g. a conical refiner or a disk refiner. The specific energy consumption in the refining process is of the order of 10 - 1000 kWh/t, preferably 100 - 700 kWh/t, most preferably 10 - 300 kWh/t.

[0023] The noil of the invention can be used in the manufacture of any kind of paper or cardboard produced on a paper or cardboard machine. The noil of the invention is especially well suited for use in the manufacture of information paper or cardboard, e.g. in the manufacture of printing paper and copying paper sorts having a grammage of the order of 25 - 350 g/m², or in the manufacture of graphic cardboard sorts in which the surface layer has a grammage of the order of 25 - 350 g/m² and

the backing or inner layer is any kind of base layer known in the art. The invention is especially applicable in the manufacture of case board, bleached kraft liner or the like. The paper pulp to be used may contain any pulp components, pigments, additives or other substances in known proportions of weight, as is generally known in paper manufacturing industry or in relevant literature.

[0024] In the following, the invention will be described in detail by the aid of a few examples of its embodiments by referring to the attached drawings, wherein

[0025] Figures 1 - 6 represent the light-scattering coefficient of the paper as a function of its tensile index, and the air permeability, interlaminar strength, CaCO₃ retention, light-scattering coefficient and density as functions of the CaCO₃ content of the pigment used, for certain test paper sorts when the noil of the invention is used together with precipitated calcium carbonate, ground calcium carbonate and without them.

EXAMPLE 1

[0026] In this experiment, printing paper was produced using a fibrous mixture containing 50 % bleached chemical softwood pulp refined to the value CSF 420 and 50 % groundwood. The noil was produced by refining chemical birch pulp in a Valley laboratory hollander in accordance with the SCAN-C 25:76 standard for 2.5 hours. The refined pulp was screened using a Bauer-McNett screen, and the P200 fraction was saved. The amount of noil added to the pulp was 4.5 w-%. The amounts of filler used were 10 w-% and 20 w-%. The noil and the filler were added into the pulp simultaneously before sheet formation. As retention agents, cationic starch in an amount of 0.65 w-% and silica in an amount of 0.15 w-% were used. From the pulps thus obtained, test sheets were produced and for each sheet the light-scattering coefficient was determined as a function of tensile index and the air permeability, interlaminar strength, CaCO₃ retention, light-scattering coefficient and density were determined as functions of CaCO₃ content.

[0027] The amounts of noil added into the pulps prepared and the pigments used in different pulp mixtures are presented in Table 1. The measurement results are presented in the form of graphs in Fig. 1 - 6.

[0028] The measurement results indicate that the addition of noil in the pulp significantly increased the tensile index value. Moreover, the addition of noil reduced the air permeability, increased the interlaminar strength, improved the CaCO₃ retention and slightly increased the density. However, the addition of noil had no significant effect on the light-scattering coefficient.

Table 1

Pulp mixture	Addition of noil	Pigment used
1	0 %	PCC

Table 1 (continued)

Pulp mixture	Addition of noil	Pigment used
2	4.5 %	PCC
3	0 %	GCC
4	4.5 %	GCC
5	4.5 %	no pigment
6	0 %	no pigment
PCC = precipitated calcium carbonate GCC = ground calcium carbonate		

[0029] The embodiment examples are intended to illustrate the invention without limiting it in any way.

Claims

1. Noil for use in paper manufacture, said noil being mixed in paper pulp, **characterised** in that the noil has been produced from cellulose fibre by refining and consists of noil fibrils which, in respect of in size distribution, mainly correspond to wire screen fraction P50, and that the noil constitutes 0.1 -15 w-% of the paper pulp.
2. Noil as defined in claim 1, **characterised** in that the noil fibrils mainly correspond to wire screen fraction P100.
3. Noil as defined in claim 1 or 2, **characterised** in that the paper pulp contains pigment, the mass ratio of pigment to noil being 0.1 - 20.
4. Noil as defined in any one of claims 1 - 3, **characterised** in that the noil has been produced by refining cellulose fibre to a Schopper number > 80.
5. Noil as defined in claim 4, **characterised** in that cellulose fibre has been refined to a Schopper number in the range 85 - 90.
6. Method for producing noil for use in paper manufacture, **characterised** in that cellulose fibre is refined so as to form noil fibrils that, in respect of size distribution, mainly correspond to wire screen fraction P50.
7. Method as defined in claim 6, **characterised** in that the noil fibrils mainly correspond to wire screen fraction P100.
8. Method as defined in claim 6 or 7, **characterised** in that the noil is produced by refining cellulose fibre to a Schopper number > 80.
9. Method as defined in claim 8, **characterised** in that cellulose fibre and/or mechanical pulp fibre is/are refined to a Schopper number in the range 85 - 90.
10. Paper pulp for use in paper manufacture, containing cellulose fibre and/or mechanical pulp fibre, filler and, if desired, other kinds of noil, **characterised** in that the paper pulp contains noil that has been produced from cellulose fibre by refining and consists of noil fibrils mainly corresponding in size distribution to wire screen fraction P50 and that the noil constitutes 0.1 - 15 w-% of the paper pulp.
11. Paper pulp as defined in claim 10, **characterised** in that the noil fibrils mainly correspond to wire screen fraction P100.
12. Paper pulp as defined in claim 10 or 11, **characterised** in that the paper pulp contains pigment, the mass ratio of pigment to noil being 0.1 - 20.
13. Paper pulp as defined in any one of claims 10 - 12, **characterised** in that the noil has been produced by refining cellulose fibre to a Schopper number > 80.
14. Paper pulp as defined in claim 11, **characterised** in that cellulose fibre has been refined to a Schopper number in the range 85 - 90.
15. Paper manufactured using noil as defined in any one of claims 1 - 5.
16. Paper manufactured using paper pulp as defined in any one of claims 10 - 14.

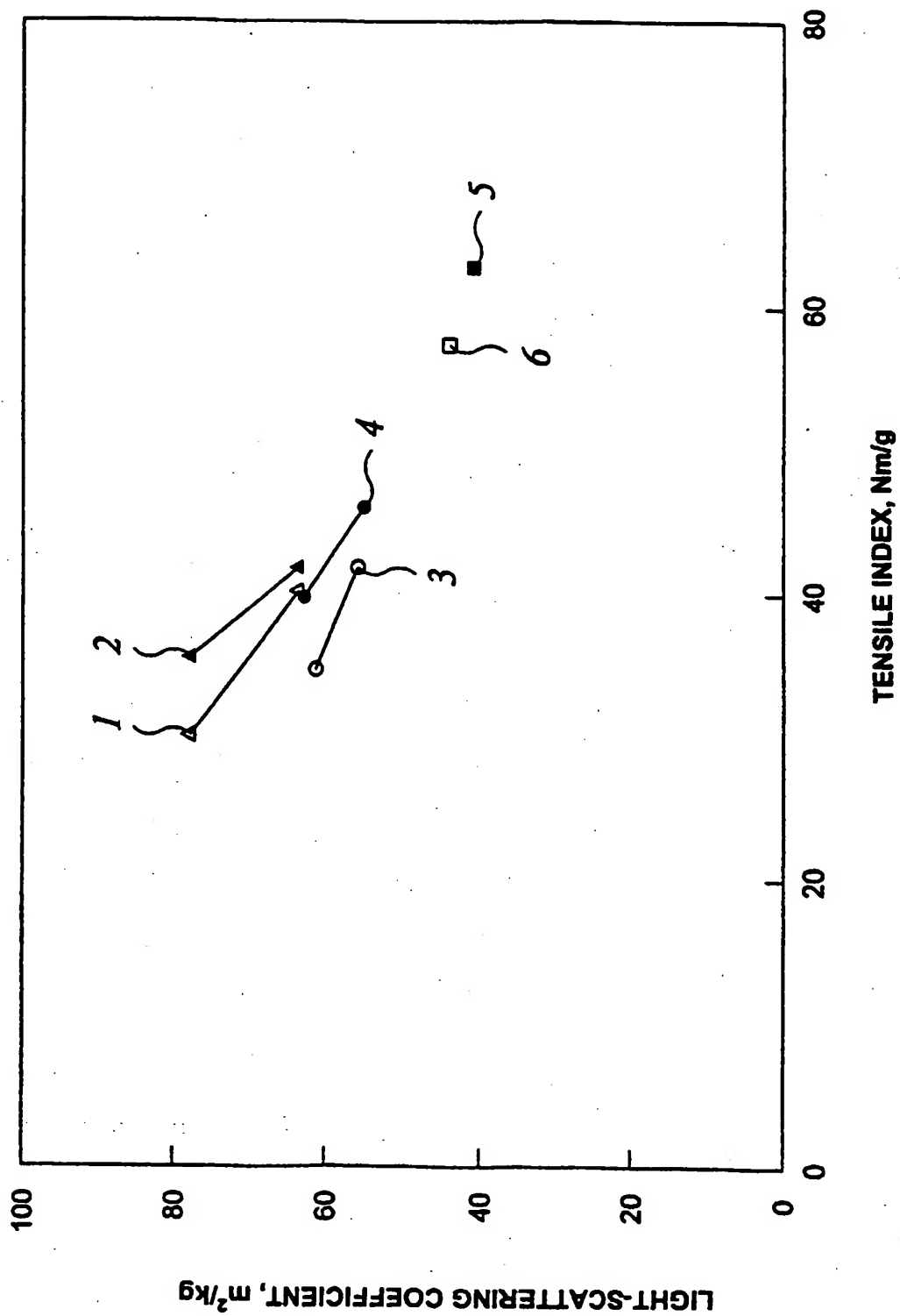


Fig. 1

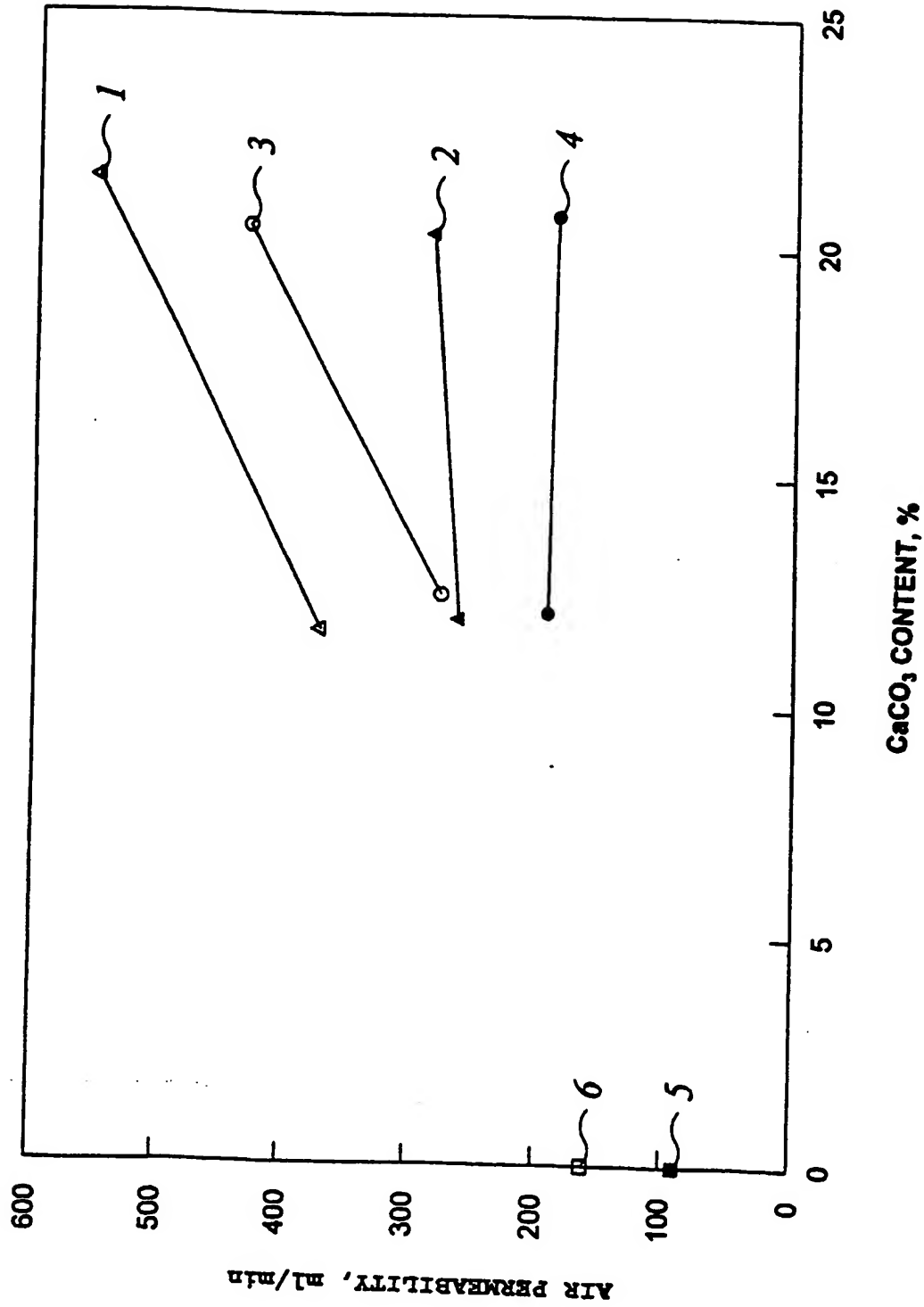
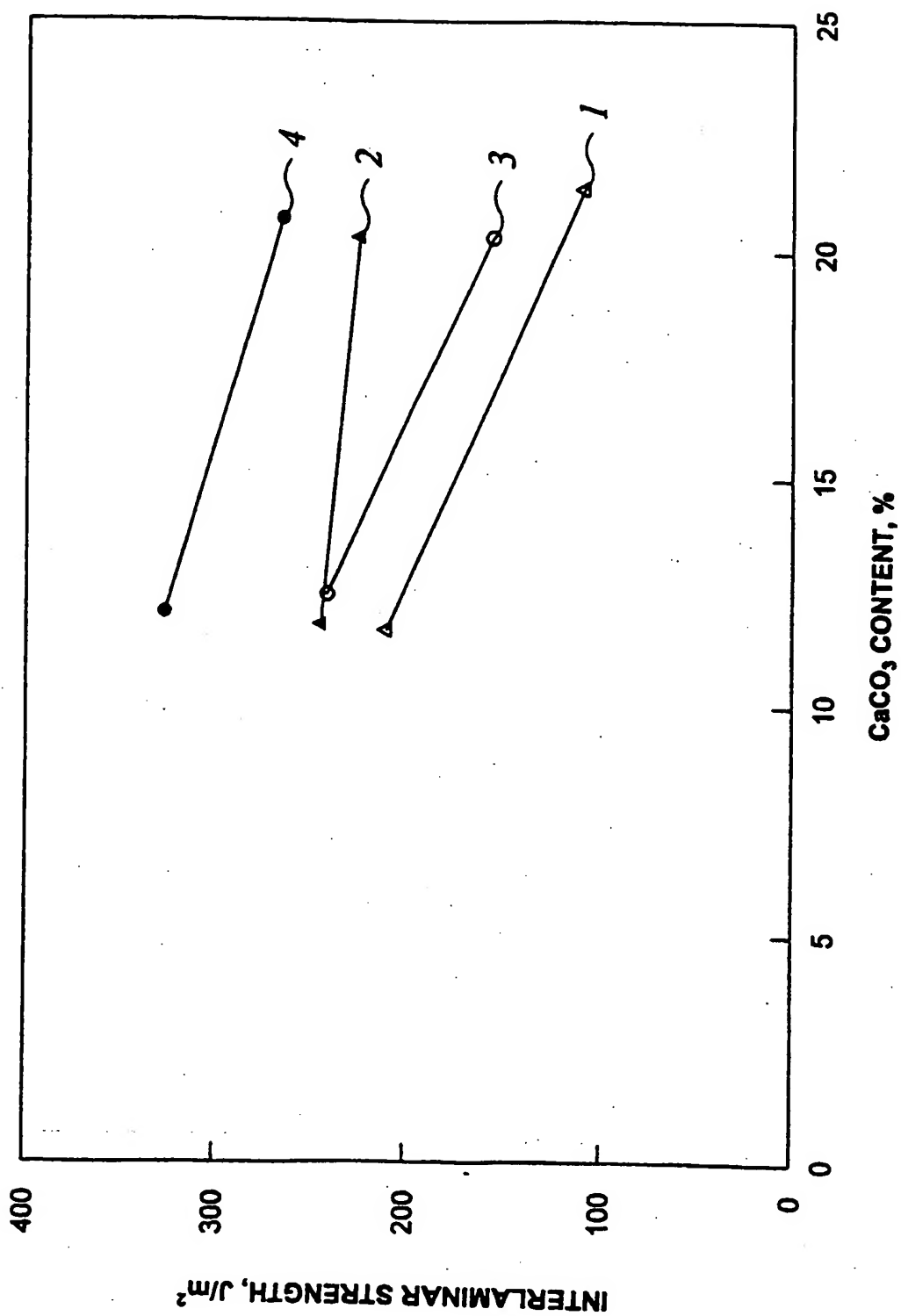
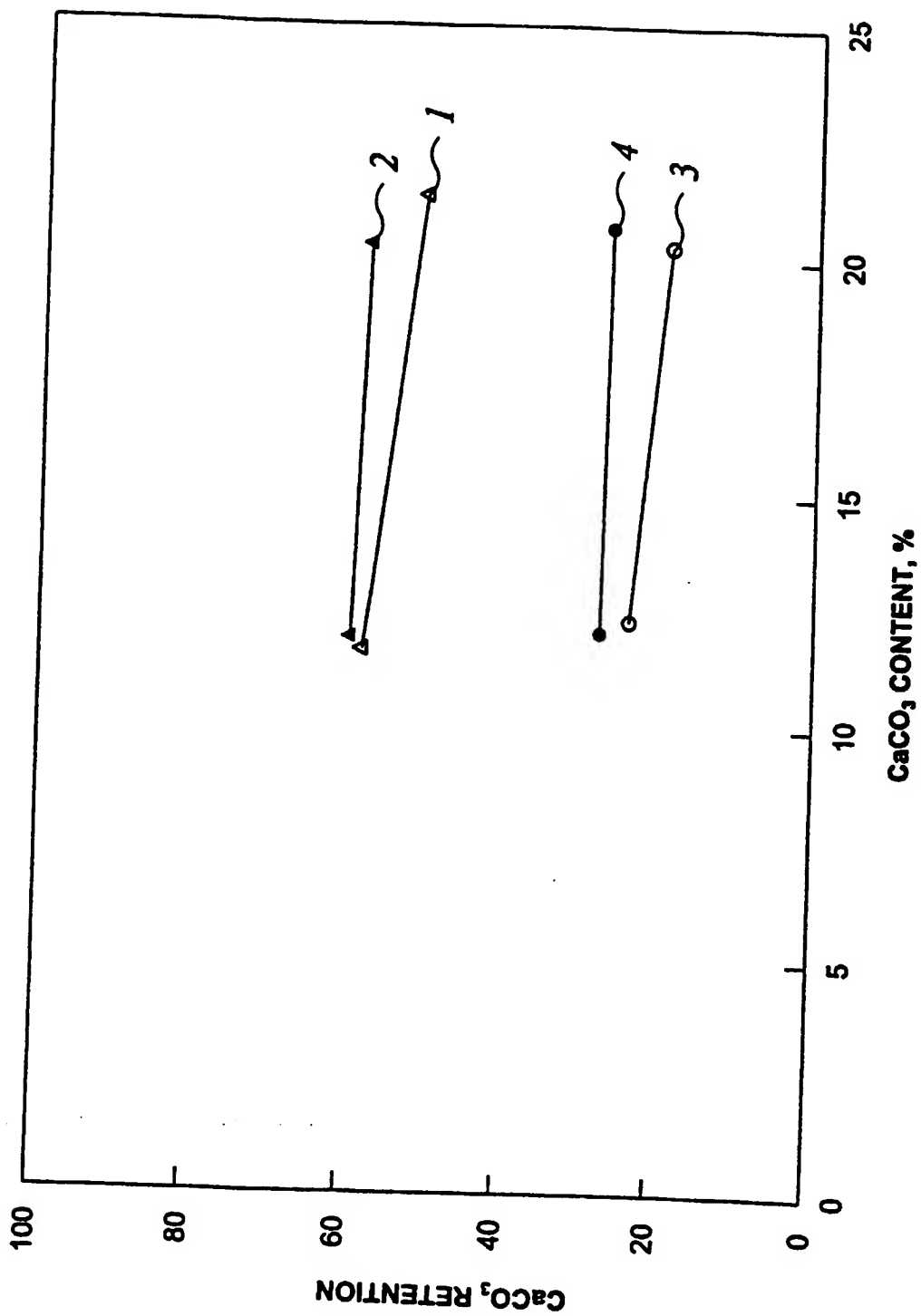
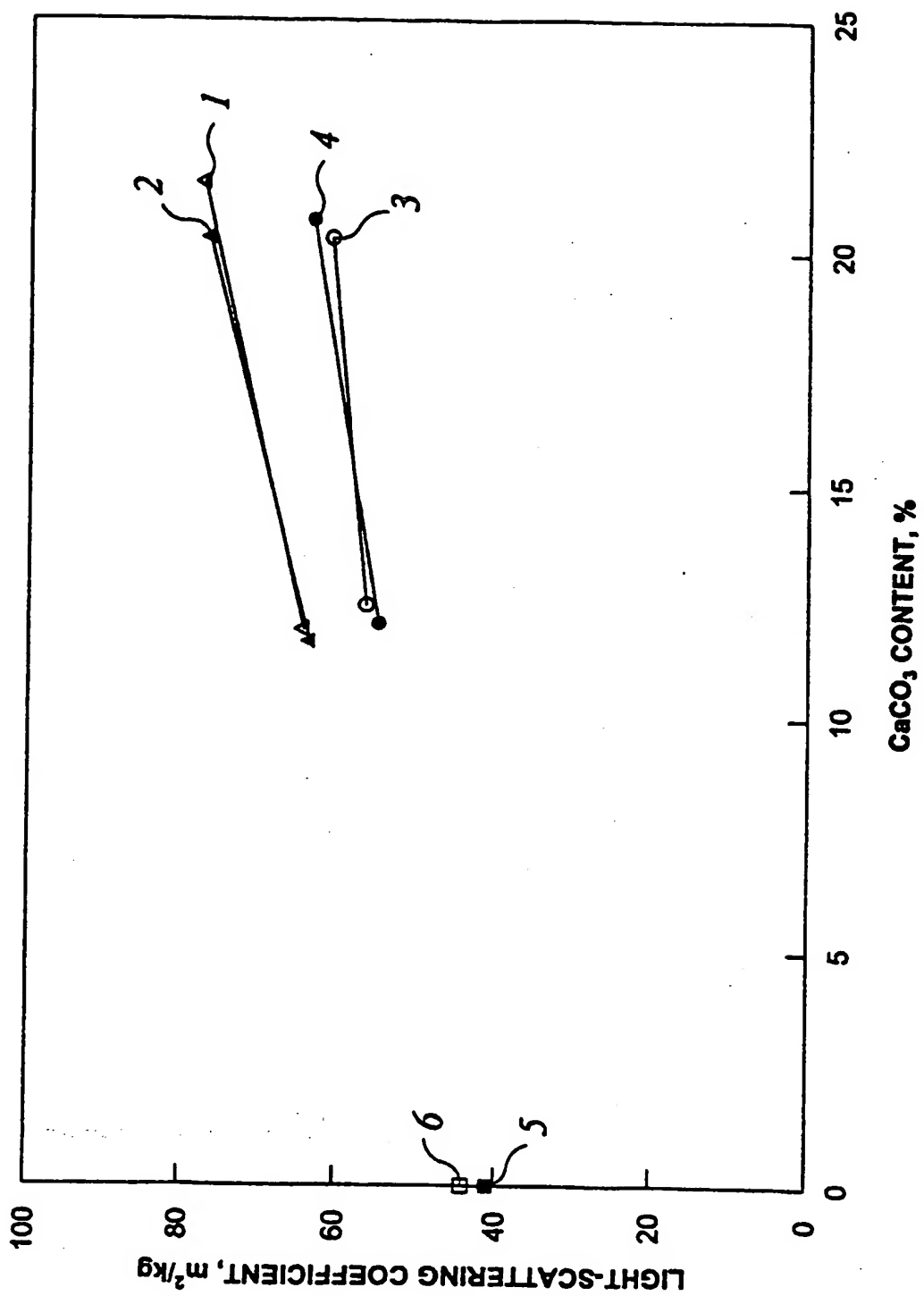
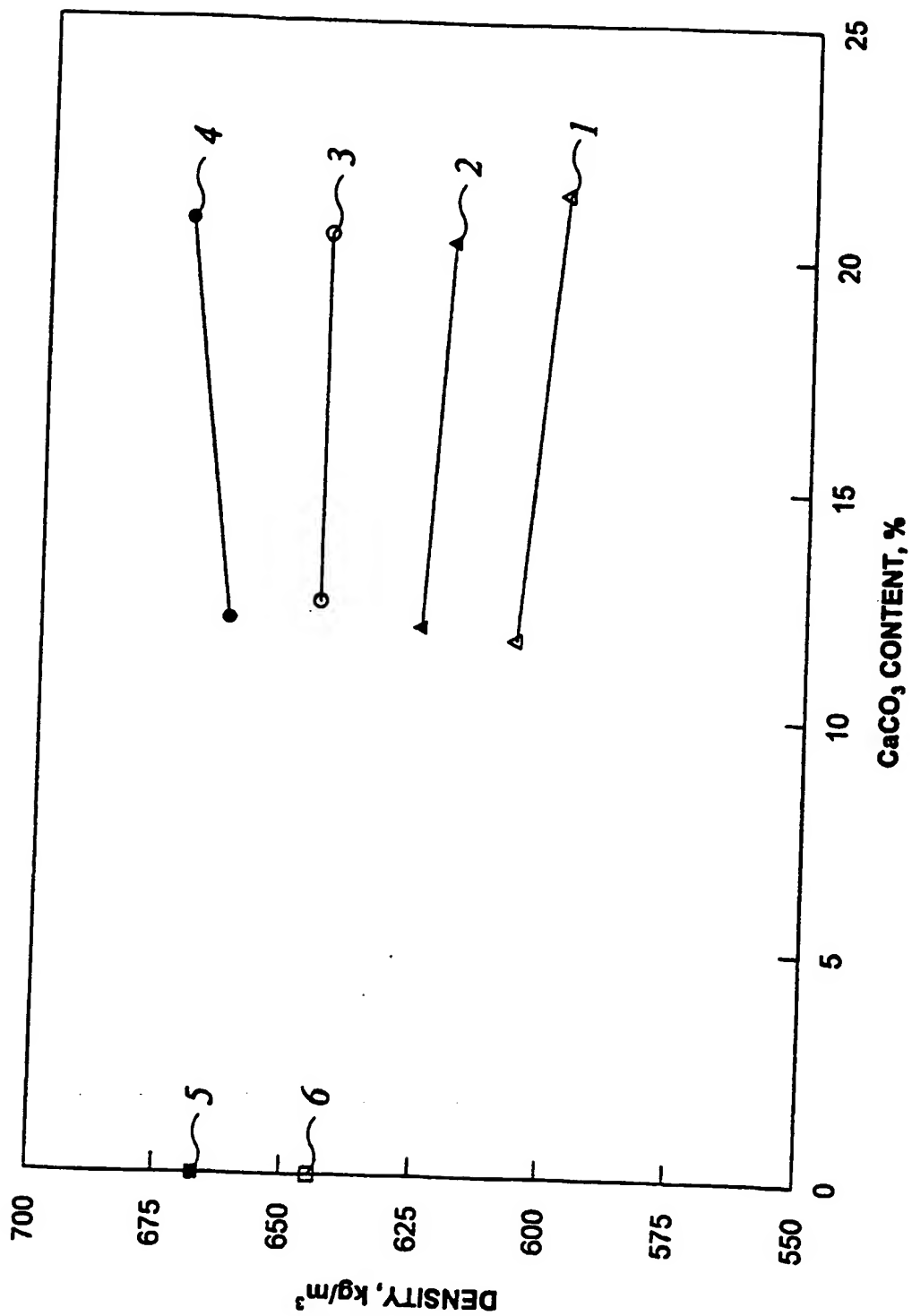


Fig. 2

*Fig. 3*

Fig. 4

*Fig. 5*

*Fig. 6*



European Patent
Office

EUROPEAN SEARCH REPORT

Application Number
EP 99 66 0003

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 8)
X	WO 97 01670 A (METSÄE SERLA OY ;SILENIUS PETRI (FI); LESKELÄE MARKKU (FI)) 16 January 1997 * example 1 *	6-9	D21H11/18 D21D1/20 D21H17/67
D	& FI 953 238 A		
X	US 4 692 211 A (ROBERTS JAMES R) 8 September 1987 * the whole document *	1-16	
Y		1,6,10, 15,16	
X	US 5 387 319 A (MORA FERNAND ET AL) 7 February 1995 * column 3, line 23 - line 37; example 1 *	6-9	
Y	* claims 1,5 *	1,6,10, 15,16	
A		1-16	
Y	J.P. CASEY: "Pulp and Paper; Chemistry and Chemical Technology, 3rd edition, vol. II, 1980, pages 826-846, 854-865, 915-920, 935-940", JOHN WILEY & SONS, NEW YORK XP002101336 * page 840 - page 844 *	1,6,10, 15,16	TECHNICAL FIELDS SEARCHED (Int. Cl. 8) D21H
A	US 5 228 954 A (ERSPAMER JOHN P ET AL) 20 July 1993 * the whole document *	1-16	
A	DE 835 255 C (KLEMM KARL H.) 31 March 1952 * the whole document *	1,3,6, 10,12, 15,16	
The present search report has been drawn up for all claims			
Place of search MUNICH		Date of completion of the search 27 April 1999	Examiner Nestby, K
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document</p>			

EPO Form 1500 (02.92 (previous))

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 99 66 0003

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

27-04-1999

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 9701670	A	16-01-1997	FI 953238 A	30-12-1996
			AU 699733 B	10-12-1998
			AU 6227096 A	30-01-1997
			CA 2223955 A	16-01-1997
			EP 0835343 A	15-04-1998
			NZ 311044 A	28-07-1998
US 4692211	A	08-09-1987	NONE	
US 5387319	A	07-02-1995	FR 2647128 A	23-11-1990
			AT 104003 T	15-04-1994
			CA 2017060 A,C	18-11-1990
			DE 69007856 D	11-05-1994
			DE 69007856 T	15-09-1994
			DK 398825 T	08-08-1994
			EP 0398825 A	22-11-1990
			ES 2055387 T	16-08-1994
			FI 94272 B	28-04-1995
			IE 63968 B	28-06-1995
			JP 3082892 A	08-04-1991
			NO 176675 B	30-01-1995
			PT 94060 A,B	08-01-1991
US 5228954	A	20-07-1993	AT 140738 T	15-08-1996
			CA 2067641 A,C	29-11-1992
			DE 69212475 D	29-08-1996
			DE 69212475 T	05-12-1996
			DK 586577 T	25-11-1996
			EP 0586577 A	16-03-1994
			ES 2090654 T	16-10-1996
			FI 935284 A,B,	03-01-1994
			GR 3021116 T	31-12-1996
			MX 9202534 A	01-11-1992
			NO 934280 A	27-01-1994
			WO 9221816 A	10-12-1992
DE 835255	C		NONE	

EPO FORM P0439

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ **BLACK BORDERS**
- ☐ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☒ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.

THIS PAGE BLANK (USPTO)